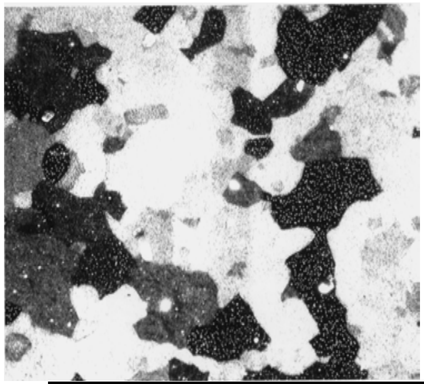
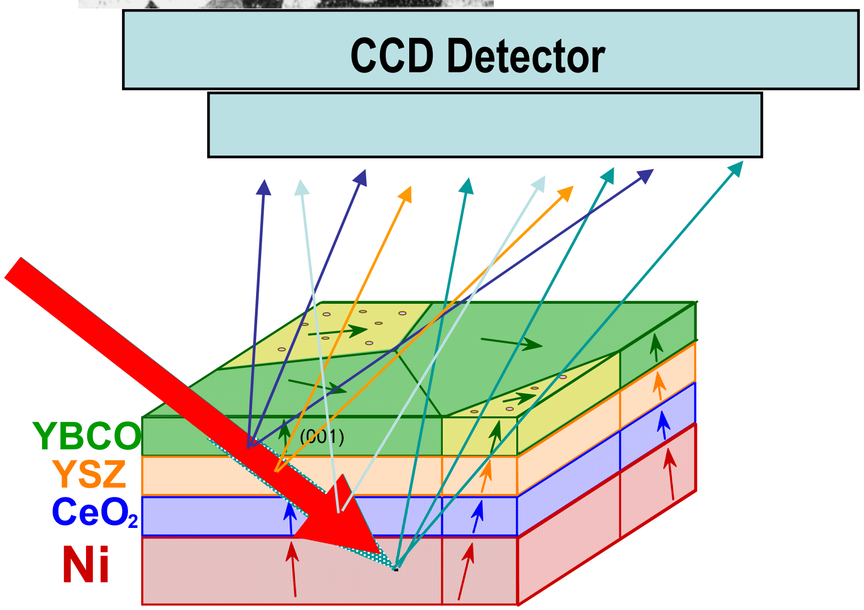
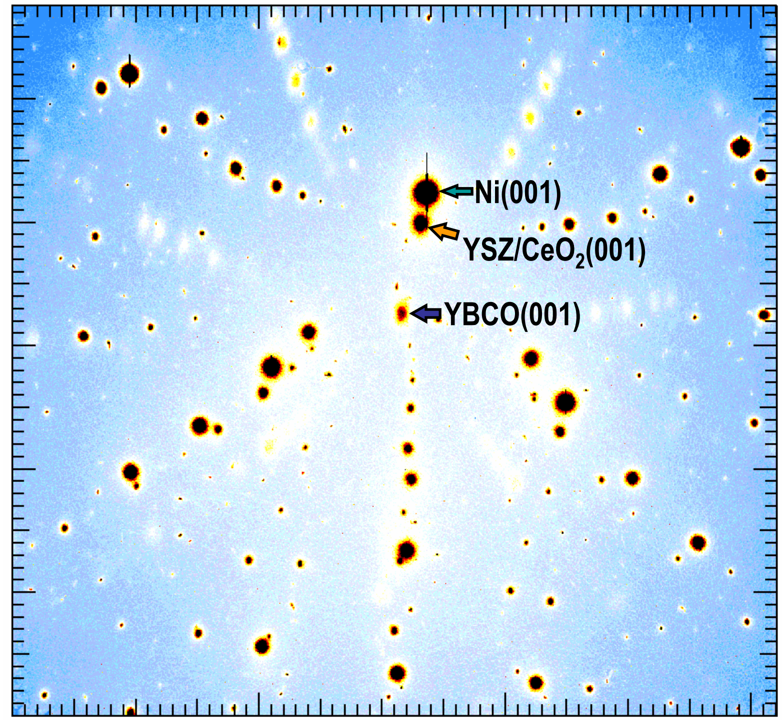
# Budai et al. polychromatic microdiffraction to epitaxial growth RABiTS

Optical: ~50$\mu$m grains

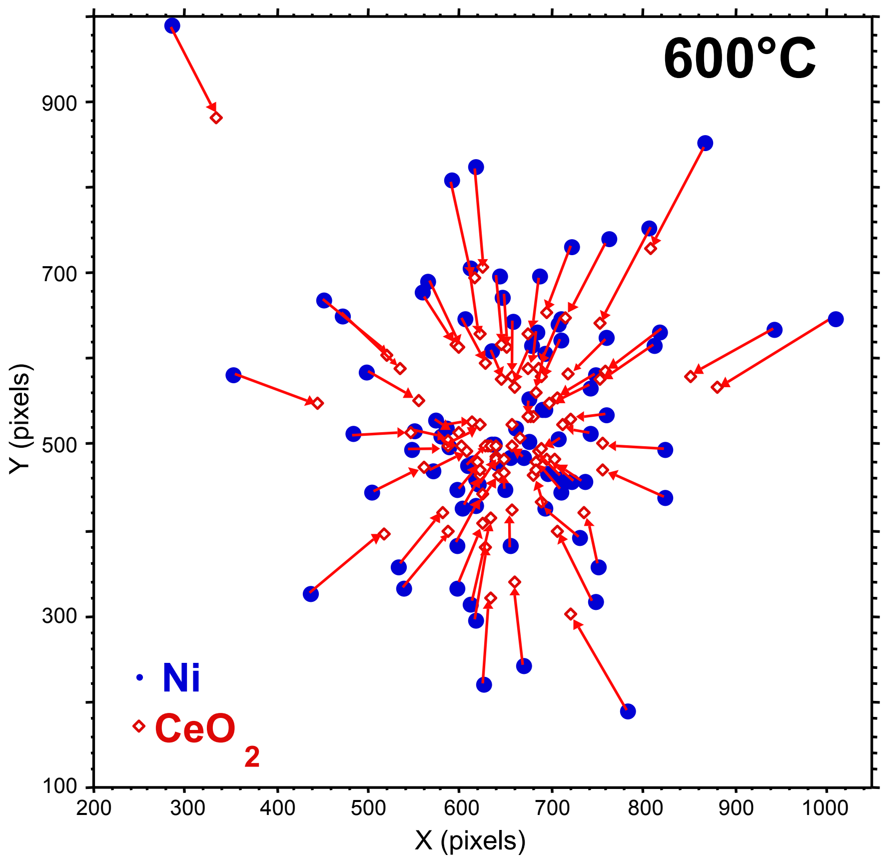


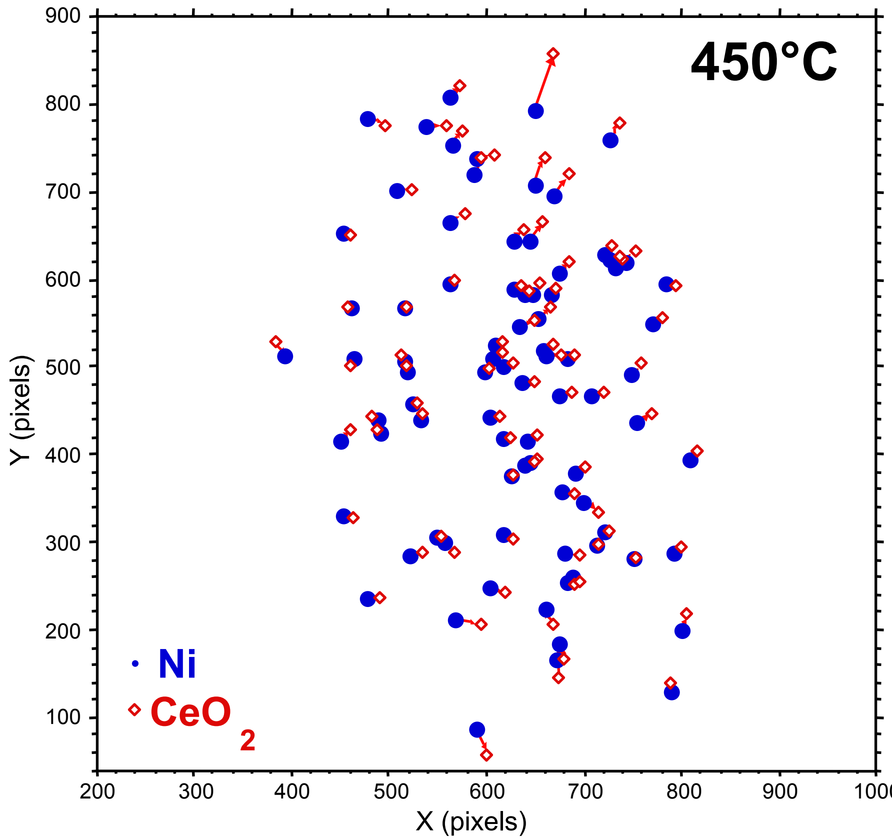



CCD Laue Patterns



# Relative $CeO_2$ orientation depends deposition temperature




**High temperature growth**:

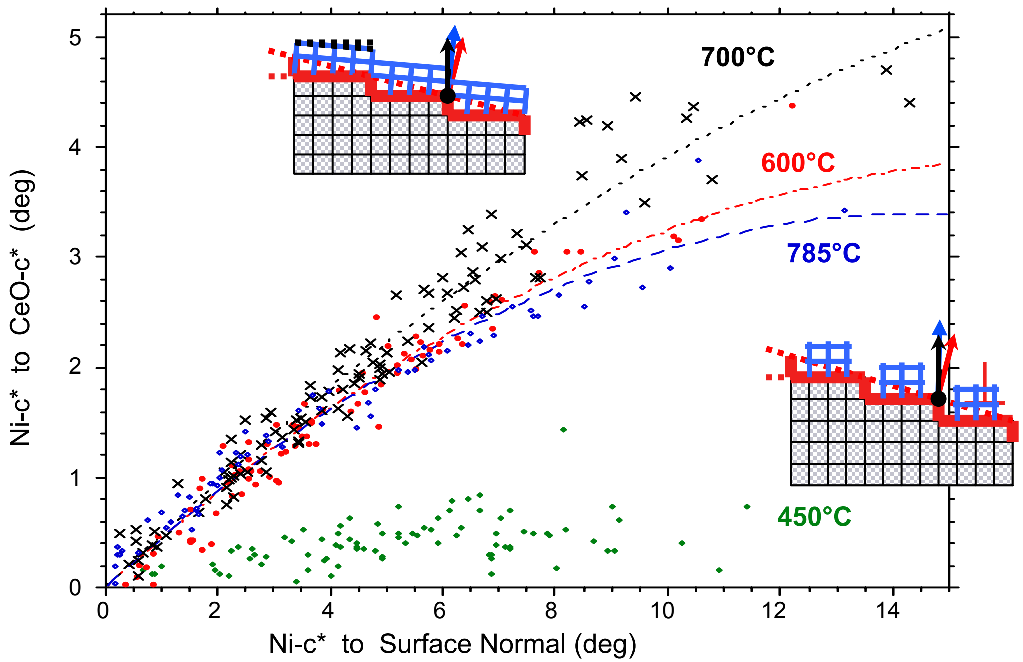
Crystallographic tilt towards ⊥

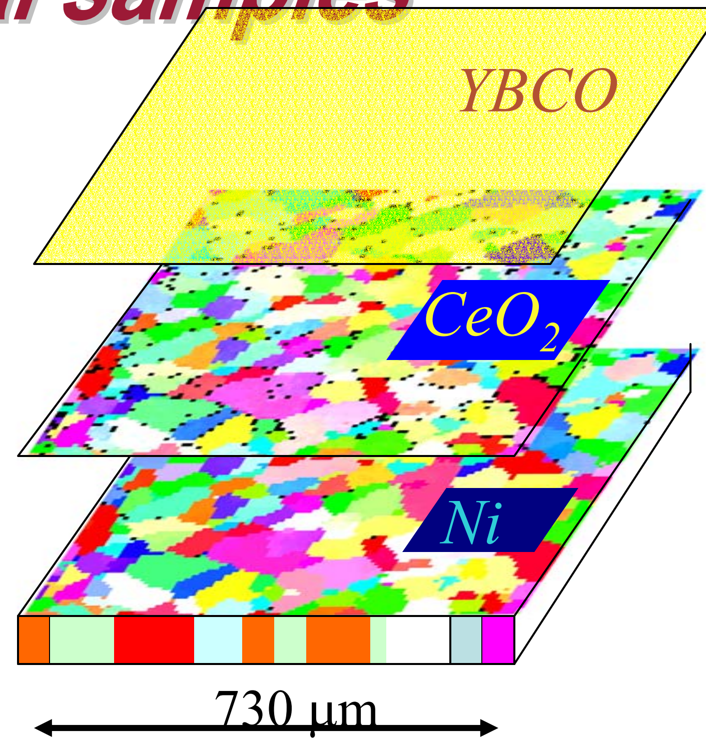
Tilt increases monotonically with miscut

**Low temperature growth**:

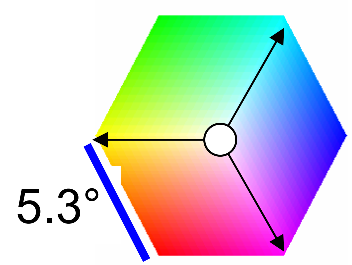
Small, ~biased tilts

# Microbeam enables combinatorial measurements on real samples



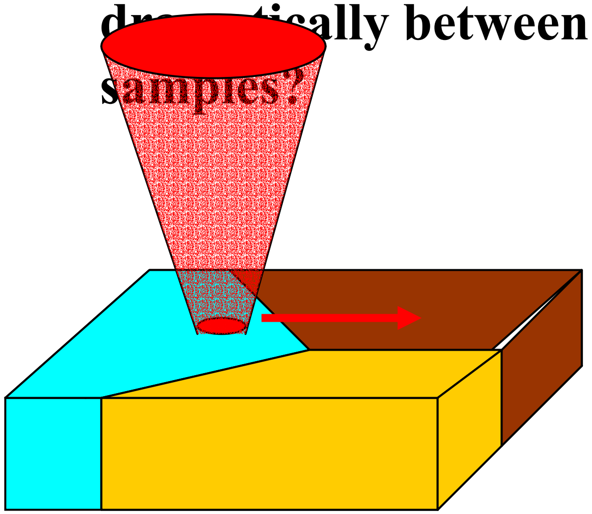



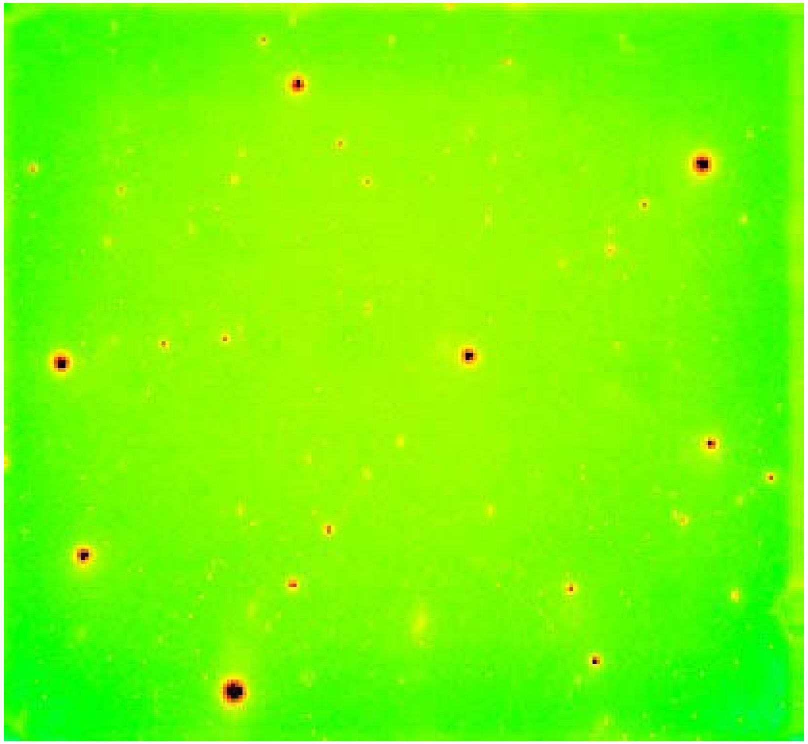

**Budai JD**, Yang WG, Tamura N, Chung JS, Tischler JZ, Larson BC, Ice GE, Park C, Norton DP **NATURE MATERIALS** 2 (7): 487-492 JUL 2003



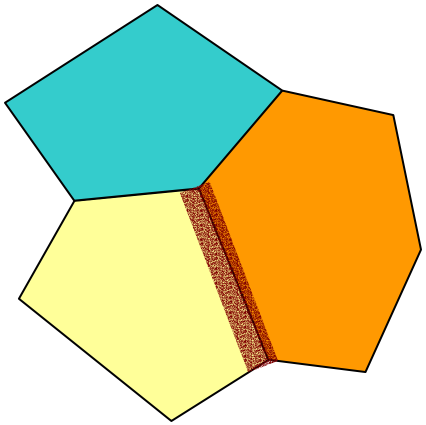
# Important questions remain

- Why does $J_c$ decrease for thick samples?
- Why does mosaic on single Ni substrate grain differ dramatically between samples?

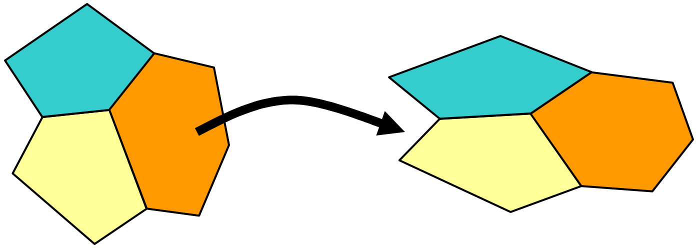




# How grain boundary/polycrystal networks interact - a major materials opportunity 21st century



- What are the constitutive equations at grain boundaries?
  - How do they change with boundary type
- What are ideal microstructures?
  - How do different networks evolve during processing and in service?
- How can grain boundary distributions be controlled?
  - Grain boundary engineering



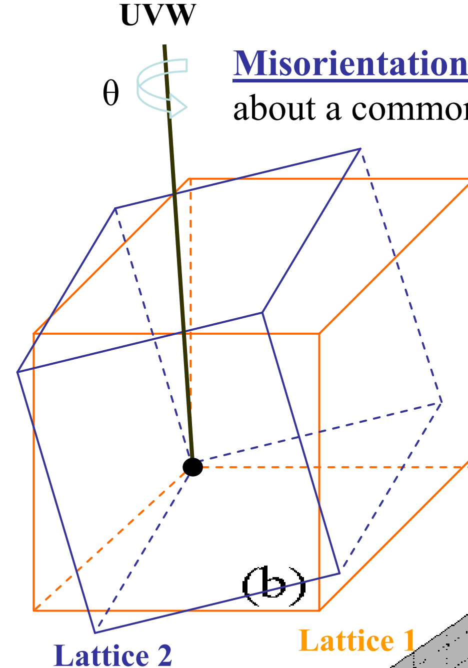
***Essential for nanophase and advanced layered materials***

# Unprecedented precision addresses long-standing issues/ tests CSL models

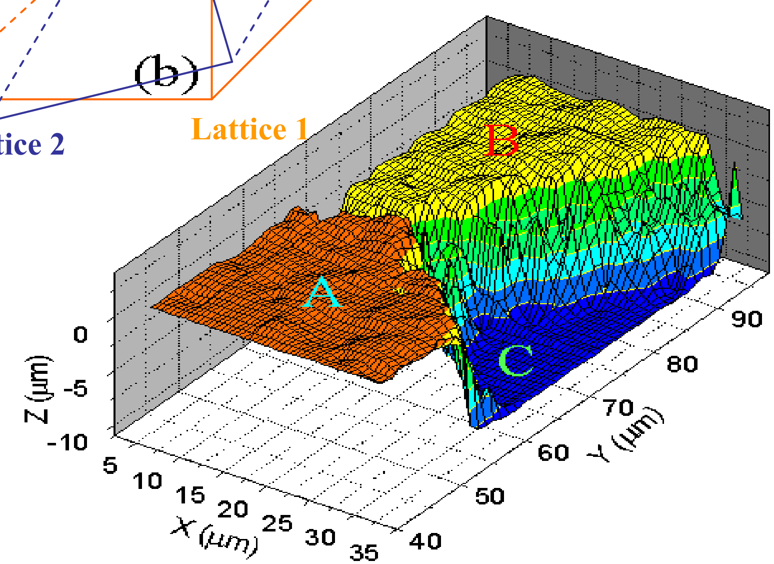
- **CSL low-energy boundaries share lattice sites**
  - **Σ denotes inverse fraction of shared sites**
  - **Theory: misorientation increases as Σ *decreases***

- **Measured misorientation increase *with Σ***

- **Grain boundary normals**
  - **Ideal directions should have lower energy**
  - **Faceting may remove energy advantage**

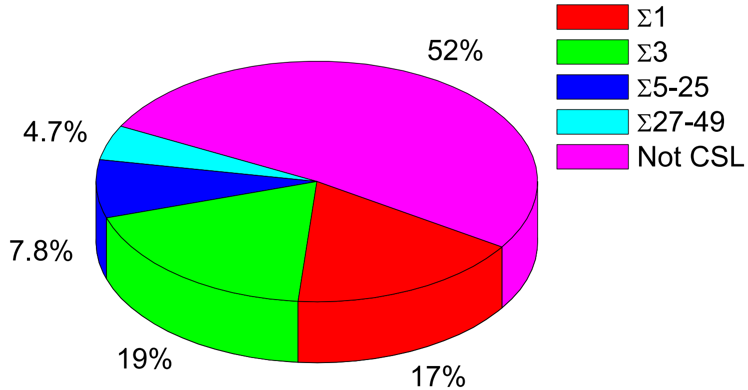



**Misorientation**: rotation of θ angle about a common axis UVW

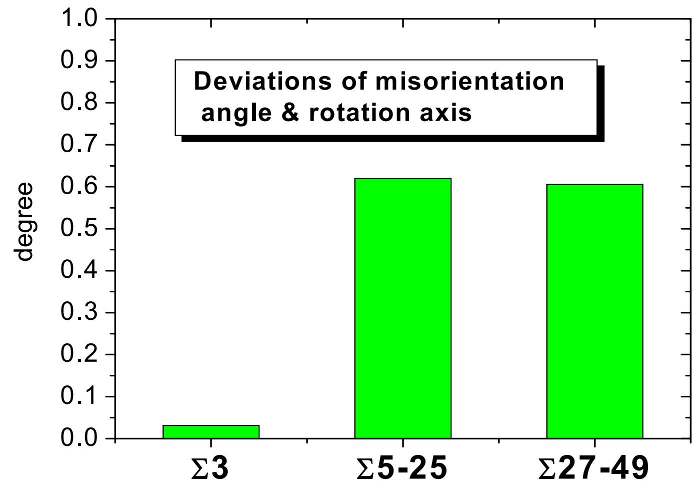



**Morphology of Ni triple junction**

# Significant statistical information emerging




Total: 70



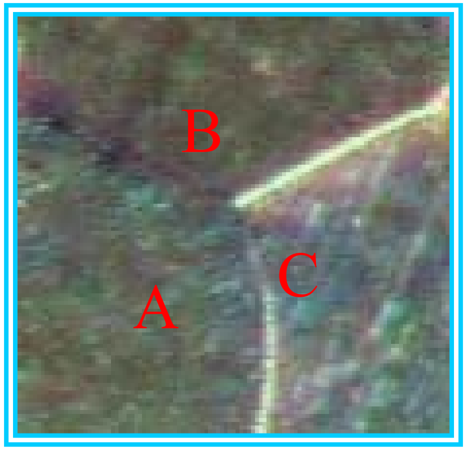

About 50% are CSLs, and 20% are found to be tilt, twist or having low-index in both grains.

| No | Sigma type | Rotation Angle (degree) | Rotation angle off (degree) | Rotation Axis (RAX) | Rotation axis off (degree) | Boundary Normal (BN) in bi-crystal | Angle between RAX – BN (degree) | |
|---|---|---|---|---|---|---|---|---|
| B2 | Σ21b | 44.40 | 0.01 | 2, 1, 1 | 2.95 | 1.00, 0.32, 0.30 / 0.69, 1.00, 0.17 | 86.3 | Tilt |
| B6 | Σ47b | 43.66 | 0.80 | 3, 2, 0 | 6.11 | 1.00, 0.07, 0.53 /1.00, 0.87, 0.31 | 74.6 | Tilt |
| B10 | Σ37c | 50.57 | 0.14 | 1, 1, 1 | 4.55 | 0.08, 1.00, 0.26 /1.00, 0.12, 0.68 | 57.6 | |
| B34 | Σ1 | (6.16°) | | | | 0.00, 1.00, 0.17 / 0.04, 0.27, 1.00 | 88.3 | Tilt |
| A57 | Σ3 | 60.00 | 0.01 | 1, 1, 1 | 0.02 | 1.00, 0.11, 0.02 / 0.32, 1.00, 0.87 | 86.4 | Tilt |
| A314 | Σ3 | 60.00 | 0.01 | 1, 1, 1 | 0.03 | 0.28, 0.31, 1.00 /0.36, 0.37, 1.00 | 2.4 | Twist |

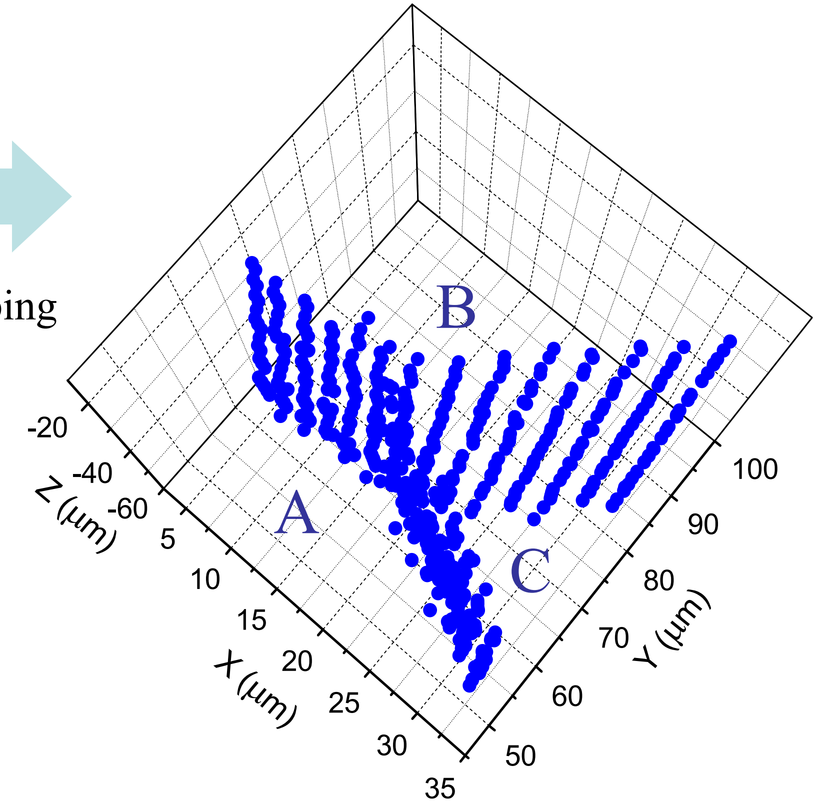
## Open questions:

1. *Why and how are the deviations from ideal CSL model as Σ type increases?*
2. *Are there residual strains imposed near the deviated CSL boundaries?*
3. *Any difference of CSLs between near or below sample surface?*
4. *…………*

# Three Dimensional Morphology of Triple Junction

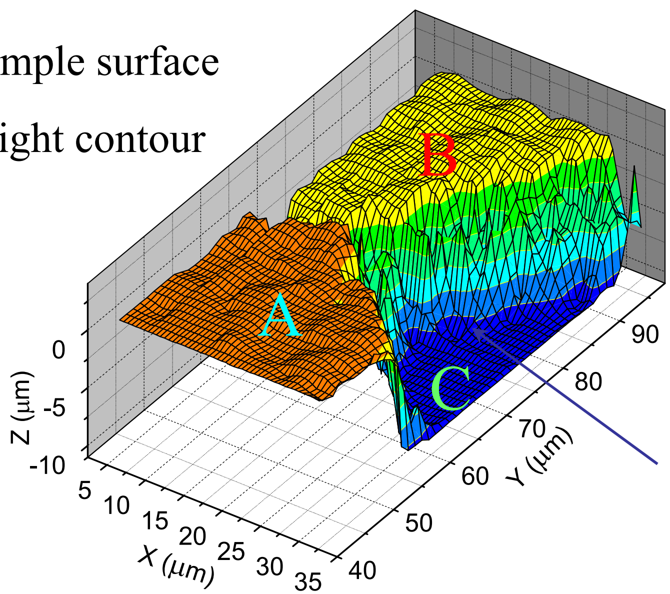



3-D mapping




Sample surface

height contour




surface step between two grains

Misorientaion angles:

A-B: 16.572°

B-C: 12.907°

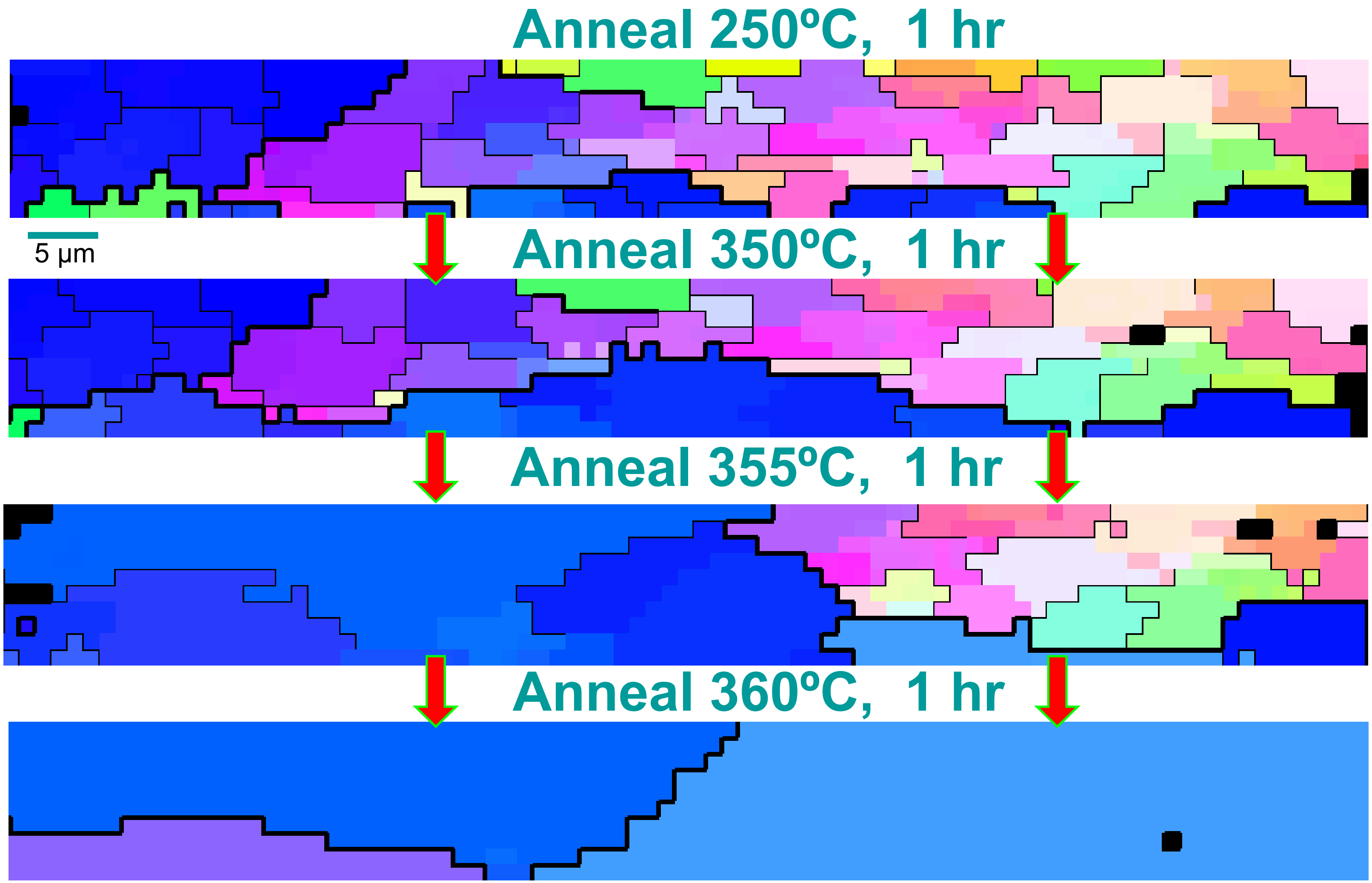
C-A: 5.538°

# Polycrystalline grain structure can now be measured nondestructively in 3D-submicron resolution-meso scale

QuickTime™ and a
Video decompressor
are needed to see this picture.

# Thermal Grain Growth in Hot-Rolled Aluminum

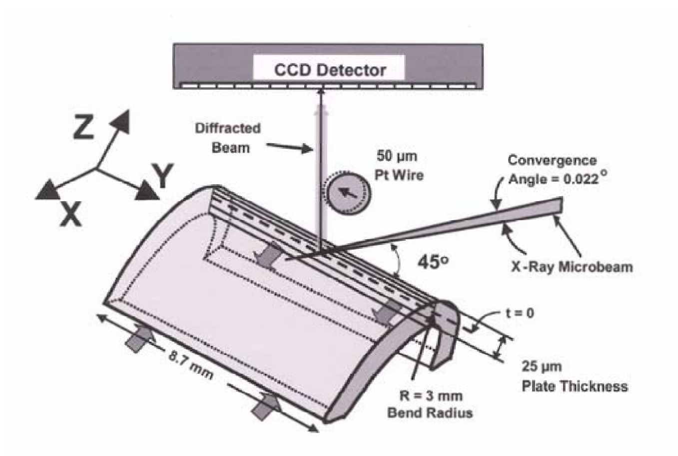
1 μm pixels, Boundaries: 5° & 20°

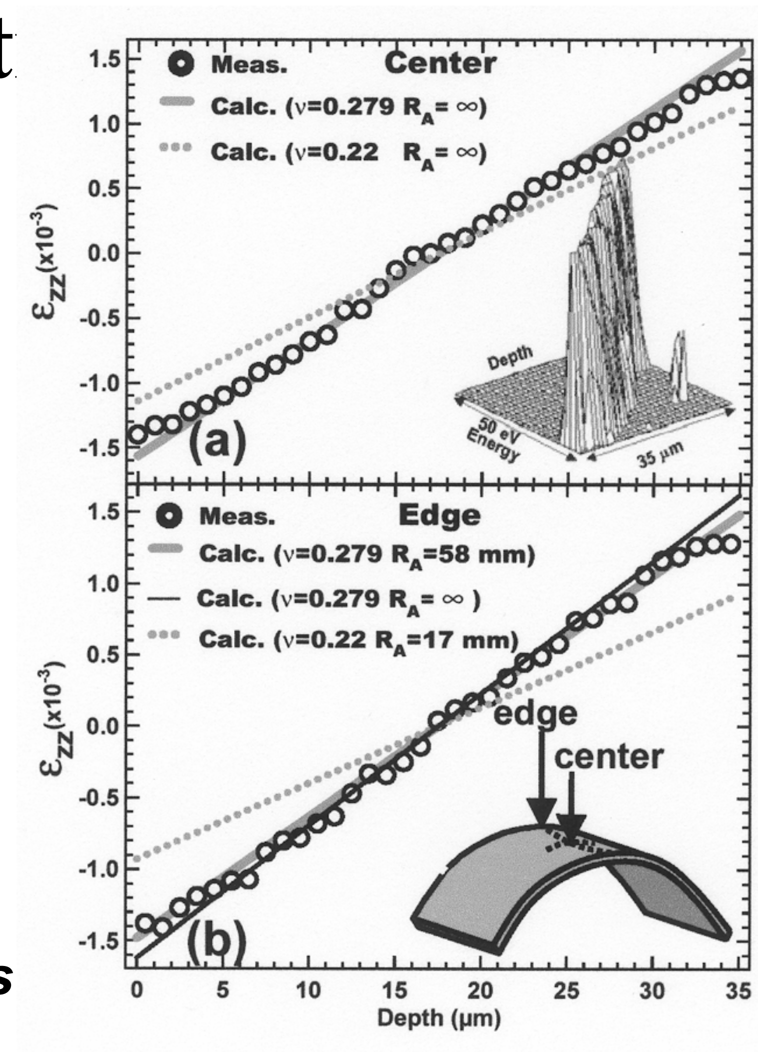



- **GB motions include both high-angle and low-angle boundaries**
- **Complete and detailed 3D evolution needed for validation of theories.**

# Elastic strain key driving force- Monochromatic DAXM measures intra-granular elast

- Local strain-even in single crystal
- Ultra-high precision local orientations
- Independent of grain orientation
- Phase sensitive

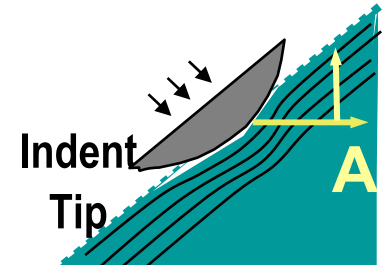



***Revolutionizes ability to study materials***

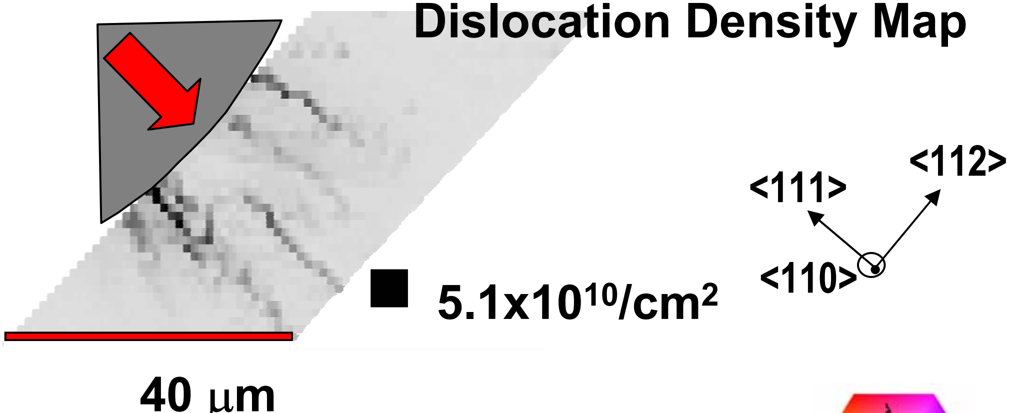
# Nanoindent in single crystals provides major insights into 3D deformation/modeling

- Deformation boundary conditions completely known/ volume modelable
- Best models predict some features not others-highly reproducible
- Single, bi-crystal, or polycrystal
- Strain-gradient models directly testable

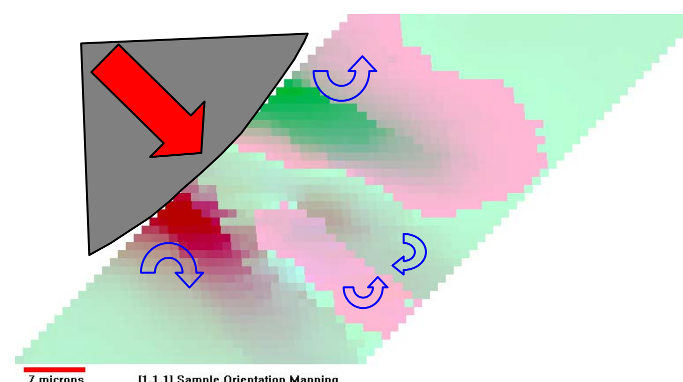


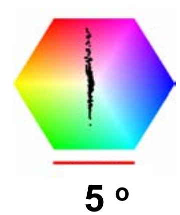

Probe Geometry

Dislocation Density Map




Lattice Rotation Map



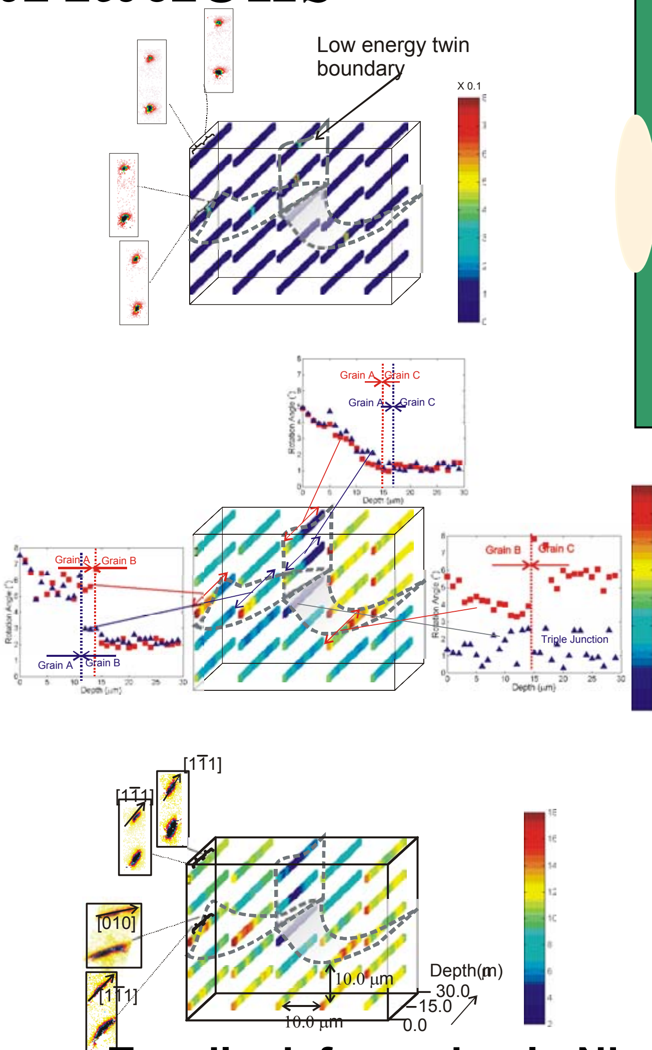

<111> Cu
100 mN Indent Force
69 μm Indent Radius

# In-situ tensile deformation polycrystal finds intra-granular variations

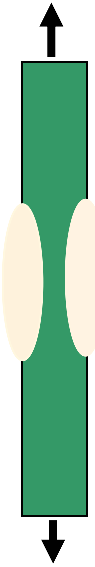
- Dramatic changes in deformations within single grain
  - Consistently large rotations near surface
- Plastic *and* elastic deformation measured
  - Essential information for understanding mechanisms
- Extensive sample characterization required for full boundary conditions

**Proposed research**

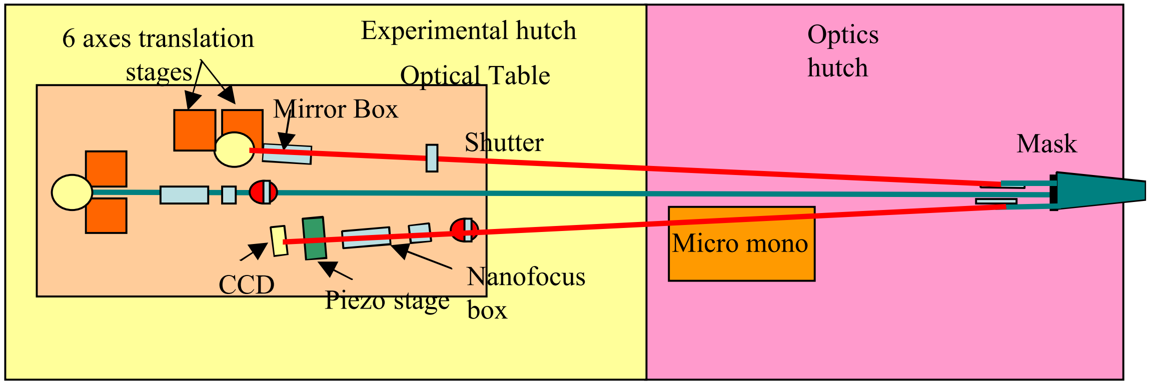
- **Full boundary conditions**
- **Low deformation**
- **Integrate theory**




**Tensile deformed poly-Ni**



# To achieve potential and meet emerging demand - new microbeam lines and hardware proposed



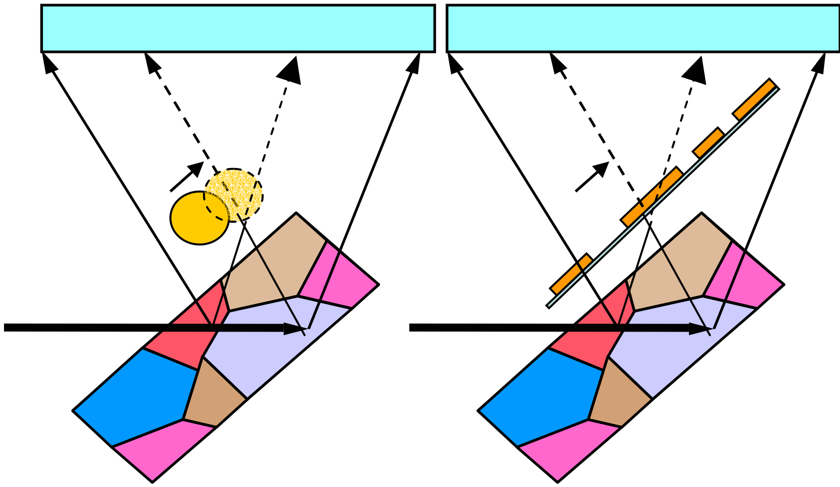

**Multiplexed 3D polychromatic diffraction-center for mesoscale research-**

- **-BM**
- **-Operated by APS**
- **-Greater general user access**

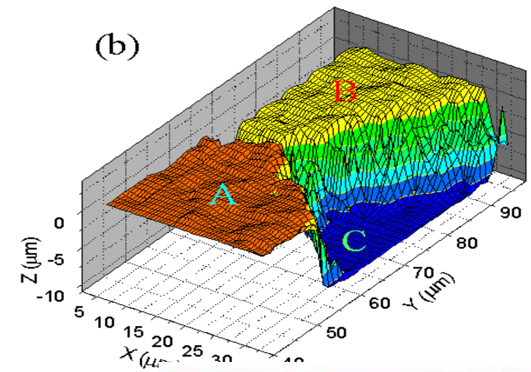
**Spatial resolution 50nm→10nm**

**Accelerated 3D characterization 100-1000x**

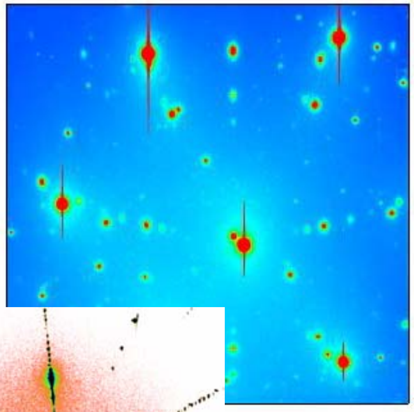
- **- Multiple wire/coded aperture**
- **- Faster detectors (GE detectors)**



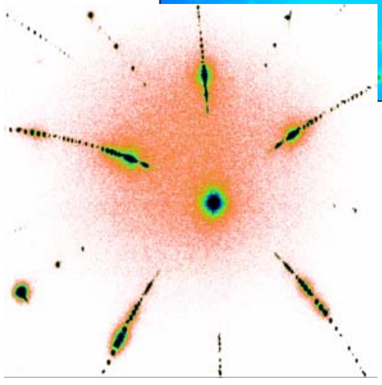
# Summary: - important emerging technique




- Cannot meet demand with existing facilities

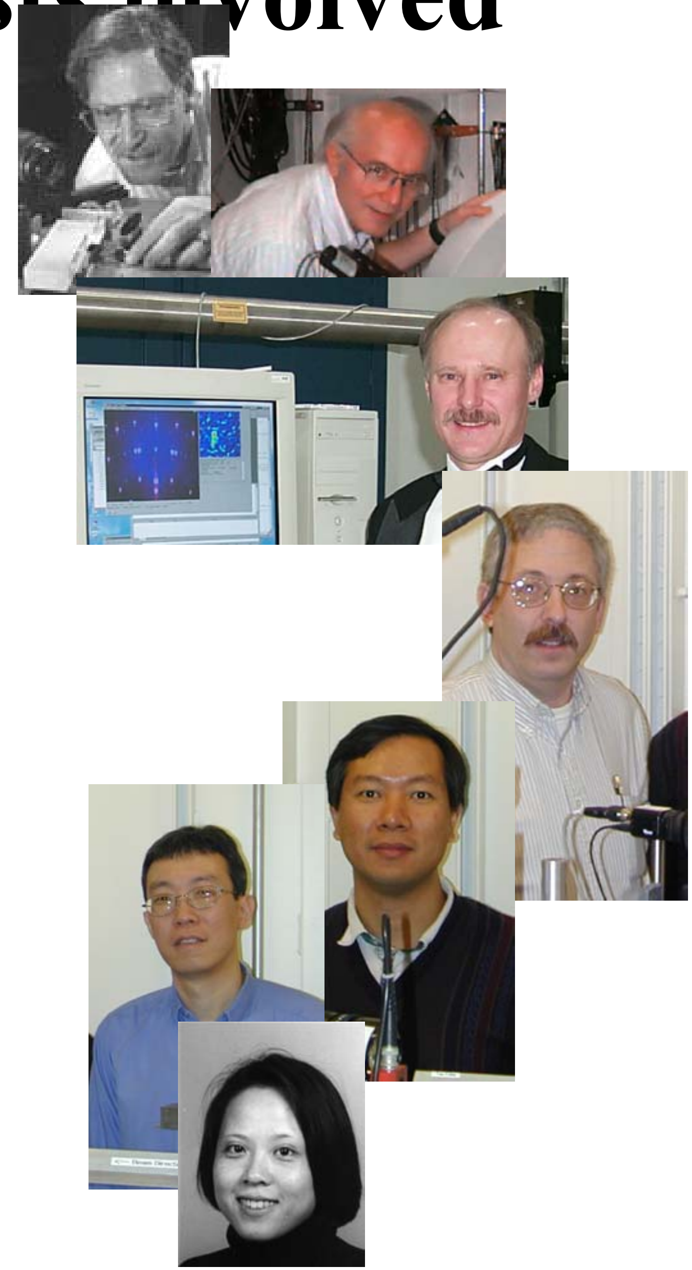


- Addresses long-standing issues with fundamentally new approach



- Wide applicability

# Team of ORNL scientists involved



- Gene Ice- **Co-principle investigator, x-ray optics**
- Bennett Larson- **Co-principle investigator-3D deformation/nanoindentation**
- John Budai-**Epitaxial films and 3D grain growth**
- Jonathan Tischler-**Mesoscale measurements and computer analysis (CMSD - APS Site)**
- Wenge Yang-**Mesoscale deformation using nanoindentation (Guest Scientist- APS Site)**
- Wenjun Liu-**Grain boundary networks (Post Doc-APS Site)**
- Judy Pang**-in-situ 3D polycrystalline deformation**

*Important support from APS-differentially deposited elliptical mirrors and beam stabilization*